



Enhanced properties of graphene/fly ash geopolymeric composite cement



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ABSTRACT

This paper reports for the first time the incorporation of in-situ reduced graphene oxide (rGO) into geopolymers. The resulting rGO–geopolymeric composites are easy to manufacture and exhibit excellent mechanical properties. Geopolymers with graphene oxide (GO) concentrations of 0.00, 0.10, 0.35 and 0.50% by weight were fabricated. The functional groups, morphology, void filling mechanisms and mechanical properties of the composites were determined. The Fourier transform infrared (FTIR) spectra revealed that the alkaline solution reduced the hydroxyl/carbonyl groups of GO by deoxygenation and/or dehydration. Concomitantly, the spectral absorbance related to silica type cross-linking increased in the spectra. The scanning electron microscope (SEM) micrographs indicated that rGO altered the morphology of geopolymers from a porous nature to a substantially pore filled morphology with increased mechanical properties. The flexural tests showed that 0.35-wt.% rGO produced the highest flexural strength, Young's modulus and flexural toughness and they were increased by 134%, 376% and 56%, respectively.

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1. Introduction

In recent years, considerable research has been aimed at the development of fly ash based geopolymers. This is driven by the need to reduce or complement the ordinary Portland cement (OPC) consumption in the construction industry. OPC is major contributor to green-house gases when compared to geopolymers. Geopolymers in general emit less green-house gases due to their lower calcium carbonate-based raw materials and production temperature. Geopolymers are inorganic polymers synthesized via a chemical reaction between a highly alkaline solution and the Si–Al minerals present in the fly ash. This results in a 3-D polymeric network consisting of Si–O–Al–O bonds with the formula of $M_n [-(SiO_2)_z - AlO_2] \cdot wH_2O$ where M is an alkaline element, n is the degree of polymerization, z is a value between 1 and 32, and w is the hydration extent, which is a function of the type and amount of the alkaline solution used [1]. The most commonly used alkaline solution is a mixture of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) with a $Na_2SiO_3/NaOH$ ratio between 1.5 and 3 [2]. The processed type F fly ash based geopolymers exhibit mechanical properties similar to those of OPC but with better performance under severe environmental conditions. For example, it has been reported that geopolymers exhibit excellent resistance to acid and sulfate attack when compared to OPC due to the lower calcium content of the fly ash [3–5]. Fly ash based geopolymers are also fire

resistant binders. According to Pan et al. [6], geopolymers are inherently fire resistant due to their polymeric-silicon–oxygen–aluminum framework. Pan et al. [6] and Duxson et al. [7] have shown that geopolymers can sustain high temperatures (up to 800 °C) with little gel structural degradation.

OPC and geopolymers are typically brittle and characterized by low tensile strength and fracture toughness. To combat this, OPC and geopolymers are often reinforced with micro- and nano-fibers. For example, fibers such as steel, polypropylene (PP), polyvinyl chloride (PVC) and basalt fibers have been found to be particularly effective in controlling crack propagation and enhancing the flexural strength and the fracture energy of geopolymers [8]. These enhanced properties were mainly attributed to the fiber bridging effect during both micro- and macro-cracking of the geopolymeric matrix [8]. The mechanical interlocking at the fiber–matrix interface and chemical bonding between the fiber and the matrix both play a role in strengthening and toughening of the geopolymeric matrix [9].

Recently, carbon nanotubes (CNTs) were adopted as a reinforcement for geopolymers. The unique properties such as high elastic modulus and tensile strength and high aspect ratio make CNTs an ideal candidate for reinforcement. Mackenzie and Bolton [10] investigated the tensile strength of potassium-based aluminosilicate (clay) geopolymers containing single walled carbon nanotubes (SWCNTs) at concentrations of 0.20, 0.25 and 0.35-wt.%. The tensile strength results were inconsistent. The tensile strength decreased, and then increased before it began to decrease again. Saafi et al. [11] studied the multifunctional properties

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of geopolymers containing multiwalled carbon nanotubes (MWCNTs). Their experimental results indicated that the incorporation of MWCNTs up to 0.5-wt.% enhanced the flexural strength, the Young's modulus, the flexural toughness and the fracture energy of geopolymers. This improvement was due to a variety of CNT strengthening and toughening mechanisms, including high resistance to crack coalescence, crack deflection at the CNT/matrix interface, inducing and bridging of multiple cracks and CNT pullout on the fractured surfaces [12].

A non-aggregated dispersion of CNTs in aqueous liquid media is a prerequisite for their use as reinforcing fillers in cement and geopolymer based composites. However, dispersion of CNTs in water is highly challenging as van der Waals forces between the CNTs create bundles and agglomerates. For CNTs, this phenomenon reduces the workability and mechanical properties of composites [13,14]. Graphene offers many benefits over CNTs, including higher surface area (due to its 2-dimensional structure) and strong mechanical interaction with the hosting matrix resulting from the wrinkling [15]. However, graphene sheets exhibit very low dispersibility in polar liquids due to their high surface area and surface energy and, as a result, they agglomerate and stick to each other, thereby reducing their reinforcement effects when agglomerated [16].

Graphene oxide (GO), the oxidized form of graphite, has been considered as a precursor for bulk-scale production of low-cost graphene-based materials [17]. GO sheets contain a large concentration of hydroxyl, epoxide, carboxyl and carbonyl functional groups [18]. These functional groups are compatible with water and therefore the GO is highly dispersible in polar liquids [19]. GO-based fillers were incorporated into various plastic and organic composites [20,21]. The improved mechanical properties of these composites were attributed to the high specific surface area and excellent mechanical properties of GO sheets [21].

In view of these outstanding properties, the integration of graphene into geopolymers can significantly improve their properties and provide them with self-sensing capabilities. The objective of this paper is to investigate the mechanical properties, chemical functional group changes and morphological changes of geopolymers containing reduced GO (rGO) sheets at a variety of loadings. The properties discussed herein are the morphology characteristics, the flexural strength, the Young's modulus, the flexural toughness and the void and pore filling mechanisms together with chemical changes associated with the alkaline reduction of GO.

2. Experimental program

2.1. Materials

Class F fly ash was used to process the rGO-geopolymeric composites. The chemical composition of the fly ash is given in Table 1. Based on the size distribution tests conducted by the supplier, 70% by weight of the total type F fly ash spherical particles was between 0.2 and 5 μm in diameter.

Hydrophilic and oxygenated 1.1 nm thick pristine GO sheets (0.5–5 μm with purity higher than 90%, supplied by Supermarket®) were employed in this study. The required GO content was dispersed in deionized water by using a 100 W Cell Disruptor for 1.5 h. The plain geopolymeric matrix had a bulk of density of 2.0 g/cm³ and composed

of 72-wt.% fly ash, 20-wt.% of sodium silicate (Na_2SiO_3 with 29.4% SiO_2 , 14.7% Na_2O and 59.9% H_2O) and 8-wt.% of 10 M sodium hydroxide (NaOH). The ($\text{Na}_2\text{SiO}_3 + \text{NaOH}$) to fly ash ratio was 0.39.

2.2. Fabrication of rGO-geopolymeric composites

To chemically reduce the GO sheets during mixing and curing, the dispersed pristine GO sheets were first added to 100 g of NaOH solution (10 M) and mildly sonicated for 1 h. The stable and heterogeneous mixture was then mixed with the fly ash and the left over chemicals ($\text{Na}_2\text{SiO}_3 + \text{NaOH}$) for 1 min. Subsequently, the mix was subjected to sonication for 3 min prior to casting the beams. Geopolymeric beams (50 mm \times 50 mm \times 350 mm) containing different concentrations of rGO (0.00, 0.10, 0.35 and 0.50-wt.%) were prepared. The molded geopolymeric beams were first cured at a room temperature of 25 °C for 2 h and then placed in an oven for 24 h at a constant temperature of 60 °C.

2.3. Morphology and chemical characterization

The morphology of the rGO-geopolymeric composites and the rGO sheets was examined with a scanning electron microscope (SEM). SEM observations were also performed on the rGO-geopolymeric suspensions in order to identify their morphology during processing. An Agilent Technologies Exoscan 4100 Fourier transform mid-infrared spectrometer, with diffuse sample interface, was used to collect infrared diffuse spectra. The instrumental conditions for spectral collection were 128 scans at a resolution of 8 cm^{-1} . The spectral changes both in terms of size and position were used to identify the processes and chemical changes in the pristine GO sheets and geopolymeric composites. In addition, the geopolymeric gels and the rGO sheets were also studied with particular attention paid to the chemical bonding and functional groups present.

2.4. Mechanical characterization

In total, 10 geopolymeric beams per rGO content were prepared for the mechanical characterization. The beams had a clear span of 210 mm and a distance between the two loading contacts of 70 mm. The beams were subjected to four-point bending tests to determine the mechanical properties; namely, flexural strength, Young's modulus and flexural toughness. The four-point bending tests were conducted under displacement control with a rate of 0.05 mm/min. During testing, load and deflection at the center were recorded continuously. The flexural strength (σ_f) and the Young's modulus (E) were calculated as:

$$\sigma_f = \frac{3Pa}{b^3} \quad (1)$$

$$E = m \frac{a(3l^2 - 4a^2)}{4b^4} \quad (2)$$

where P is the maximum applied load, l is the length of the beam between the supports, a is the distance between the support and the loading point, b is the width and thickness of the beam and m is the slope of the tangent to the straight-line portion of the load-deflection curve.

The flexural toughness of the beams is the total area under the stress-strain curve obtained from Eqs. (1) and (3), where Eq. (3) is given by:

$$\varepsilon = \frac{12b\Delta}{3b^2 - 4a^2} \quad (3)$$

Table 1

Main chemical composition of fly ash (wt.%) (as provided by the supplier).

Element	Weight %
Silicon dioxide, SiO_2	53.50
Aluminum oxide, Al_2O_3	34.30
Iron oxide, Fe_2O_3	3.60
Calcium oxide, CaO	4.40
Loss of ignition	2.00

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